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AUTHORITY

USNWC ltr, 30 Aug 1974

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IDEP FORM 12:11:02 Q4232 **SPECIFICATION** 1 of 1

1. COMPONENT/PART NAME PER GENERIC CODE: Propulsion Parts & Materials, Solid Fuel Engines	2. PROGRAM OR WEAPON SYSTEM: Multiple	D0716			
4. ORIGINATOR'S SPECIFICATION TITLE: Purchase Description - Carbon Black	5. ORIGINATOR'S SPEC. NO.: WS 7655	3. DATE OF:	DAY	MO.	YR
		ISSUE	13	10	67
		REVISION			

6. SPECIFICATION IS: ☐ DRAFT ☐ PRELIMINARY ☒ FINAL

7. THIS SPECIFICATION COMPLEMENTS REPORT NO:

8. TYPE OF SPECIFICATION

- ☒ (A) GENERAL PRODUCT REQUIREMENTS FOR A FAMILY OF PARTS - PROCUREMENT DOCUMENT
- ☐ (B) INDIVIDUAL DETAIL PARTS DOCUMENT; STDS BOOK PAGES - FOR PROCUREMENT
- ☐ (C) DETAIL INSPECTION, PROCESS CONTROL, AND/OR TEST PROCEDURES FOR SPECIFIC PARTS
- ☐ (D) PROCESS (PAINTING, WELDING, FINISHING, HEAT TREATING ETC.) APPLICABLE TO MANY PARTS
- ☐ (E) SPEC. FOR PERFORMANCE, RELIABILITY, AND/OR ENVIRONMENT FOR ASSEMBLIES, EQUIPMENTS, SUBSYSTEMS AND SYSTEMS
- ☐ (F) PERFORMANCE AND APPLICATION DATA FOR DESIGN ENG. USE ON PARTS - NOT FOR PROCUREMENT
- ☐ (G) OTHER (DETAIL IN 10.)

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1961 Book of ASTM Std. Part 20 & 28			x	

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JAN 27 1969

STATEMENT #5 UNCLASSIFIED

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13. SPEC. NO. 565.70.00.00-X7-02

11. SIGNED R. S. Harper "by" 12. CONTRACTOR NWC/CL SUBCONTRACTOR

18 50 18

NOTICES PAGE

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Code Ident
30003

WS 7655

NAVAL AIR SYSTEMS COMMAND

DEPARTMENT OF THE NAVY

PURCHASE DESCRIPTION

CARBON BLACK

1. SCOPE.

1.1 Scope. This purchase description covers one grade of carbon black, commercially known as channel black.

2. APPLICABLE DOCUMENTS.

2.1 The following documents of the issue in effect on date of invitation for bids or request for proposal form a part of this document to the extent specified herein.

SPECIFICATIONS

Federal

RR-S-366	Sieves; Standard, for Testing Purposes.

FSC 6810

STANDARDS

Military

MIL-STD-129	Marking for Shipment and Storage.

(Copies of specifications, standards, drawings, and publications required by suppliers in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

2.2 Other publications. The following documents form a part of this document to the extent specified herein. Unless otherwise indicated, the issue in effect on date of invitation for bids or request for proposal shall apply.

American Society for Testing and Materials

1961 Book of ASTM Standards; Part 28	Test-Method ASTM D1512-60, "pH Value of Carbon Black".
1961 Book of ASTM Standards; Part 28	Test-Method ASTM D1620-60, "Volatile Content of Carbon Black".
1961 Book of ASTM Standards; Part 28	Test-Method ASTM D1520-65, "Iodine Absorption Number of Carbon Black".
1961 Book of ASTM Standards; Part 20	Test-Method ASTM D1483-60, "Oil Absorption of Pigments by Gardner-Coleman Method".

(ASTM Publications are published by the American Society for Testing and Materials, Philadelphia 3, Pennsylvania.)

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3. REQUIREMENTS.

3.1 Preproduction sample. Unless otherwise specified (see 6.2), a preproduction sample shall meet all requirements of this document. The preproduction sample shall be prepared using the same methods and procedures proposed for production. Any production prior to acceptance of the preproduction sample shall be at the risk of the supplier.

3.2 Data. No data is required by this document or by referenced documents in section 2 unless specified in the contract or purchase order.

3.3 Compliance to documents. Carbon black shall conform to the requirements herein and to the applicable requirements of documents listed in section 2.

3.4 Product characteristics and performance. When tested in accordance with 4.7 of this document, carbon black shall meet the following product characteristics and performance.

3.4.1 Chemical and physical analysis. The chemical and physical analysis of the material shall be specified in Table I.

Table I. Chemical and Physical Analysis

Characteristic	Minimum	Maximum
Carbon black, %	98.0	---
Moisture, %	---	2.0
Sulfur, %	---	0.5
Acetone extract, %	---	0.5
Organic dyes, %	None	None
Ash, %	---	0.2
Acidity (hydrogen ion concentration) pH	4	6
Water soluble material	---	0.02

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Table I. (Continued)

Characteristic	Minimum	Maximum
Coarse particles (retained on a U.S. Standard No. 325 sieve)	---	0.2
Grit	---	0.1
Volatile material	---	0.1
Iodine value (milligrams of iodine absorbed per gm of carbon black)	60	75
Benzene solubles	---	0.5
Bulk density, gm/cc	0.14	0.22
Oil absorption, lbs/100 lbs carbon black	35	45

3.5 Workmanship. The carbon black shall be uniform in quality, free from foreign materials, and shall be manufactured under conditions and procedures standard in the industry.

4. QUALITY ASSURANCE PROVISIONS.

4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified, the supplier may utilize his own facilities or any commercial laboratory acceptable to the Government. The Government reserves the right to perform any of the inspections set forth in this document where such inspections are deemed necessary to assure that supplies and services conform to prescribed requirements.

4.2 Lot. A lot shall consist of material produced at one plant with no change in formulation or process. If manufacture is by batch process, each batch shall constitute a lot. A batch shall be as defined in 6.3.

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4.3 Acceptance sampling. The number of containers to be chosen at random for acceptance sampling shall be equal to the square root of the total number of containers in the lot. If the number thus obtained is not a whole number, the number of containers to be sampled shall be increased to the next higher whole number. In no case, however, shall the number of containers to be sampled be less than seven (unless there are less than seven containers in the lot, in which case, each container shall be sampled).

4.3.1 Primary sample. From each selected container, a sample shall be taken from three or more places throughout the container. The total weight of the samples taken from each container shall weigh at least 50 grams (gm). Each sample thus taken shall be mixed thoroughly, placed in a clean dry container, and labeled to identify the material name, original container designation, contract number, and lot number.

4.3.2 Composite sample. Each primary sample shall be subdivided to prepare a composite sample (not in excess of 200 gm). Primary material not used shall be returned to the primary sample container. After mixing the composite sample thoroughly, the composite sample shall be placed in a clean, dry container and sealed. The composite sample shall be identified with the material name, container designation, contract number, and lot number. All specified chemical tests shall be made on this composite sample representing the lot. Failure of the composite sample to pass all of the tests herein shall result in rejection of the lot represented.

4.4 Classification of tests. Inspection and testing of carbon black shall be classified as follows:

(a) Preproduction tests.

(b) Quality conformance tests.

4.5 Preproduction tests. Preproduction test shall be conducted only on the preproduction sample and shall consist of all examinations and tests specified in 4.6.

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4.6 Quality conformance tests. Quality conformance tests for acceptance of the carbon black shall consist of the following tests:

Characteristics	Test
Carbon black	4.7.1
Moisture	4.7.2
Sulfur	4.7.3
Acetone extract	4.7.4
Organic dyes	4.7.5
Ash	4.7.6
Acidity	4.7.7
Water soluble material	4.7.8
Coarse particles	4.7.9
Grit	4.7.10
Volatile material	4.7.11
Iodine value	4.7.12
Benzene solubles	4.7.13
Bulk density	4.7.14
Oil absorption	4.7.15

4.7 Tests. The following procedures shall be used to determine that the requirements of this document have been met. Any proposed change in test procedures or equipment shall necessitate, before adoption, prior approval of the procuring activity. In case of dispute between the results from any proposed method or equipment and what is cited herein, the results using the methods and the equipment specified in this document shall prevail. Unless otherwise specified, all tests shall be run in duplicate. The average of the two results shall be taken as the test result.

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4.7.1 Carbon black content. The carbon black content shall be determined by adding moisture content and ash content and subtracting from 100 percent.

4.7.1.1 Acceptance criteria. For the lot represented to pass the carbon black test, the value obtained for percent carbon black shall not be more than the value specified in 3.4.1.

4.7.2 Moisture determination. Add approximately 2 gm of sample to a previously dried and tared dish with ground-glass cover and weigh to the nearest milligram (mg). Place the dish and sample in an oven at 100 to 105 degrees centigrade (°C) with cover removed and heat for 2-1/2 to 3 hours. Place in a desiccator, allow to cool until a constant weight is obtained, and weigh to the nearest milligram. Calculate the loss in weight as percent moisture by the following formula:

$$\text{Percent moisture} = \frac{(A-B) \times 100}{C}$$

where: A = Weight of dish and sample before heating, gm

B = Weight of dish and sample after heating, gm

C = Weight of dish and sample minus weight of dish, gm

4.7.2.1 Acceptance criteria. For the lot represented to pass the moisture test, the value obtained for percent moisture shall not be more than the value specified in 3.4.1.

4.7.3 Sulfur determination. Transfer an accurately weighed portion of approximately 0.5 gm of the sample to the cup of a Parr sulfur bomb. Add approximately 1 gm of potassium perchlorate and mix thoroughly with a glass rod. Add approximately 15 gm of sodium peroxide, cover the cup, and mix by shaking. Assemble and tighten the bomb and place in the holder within a satisfactory shield. Ignite the contents of the bomb either electrically or by directing the flame of an oxygen-gas burner against the bottom of the cup. After the initial ignition continue the heating until the lower half of the cup attains an even cherry-red color. Remove the

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bomb from the shield, allow to stand for approximately 5 minutes and then cool in water. Remove the outer fastenings without disturbing the cap, wash the exterior of the cap and cup with water and discard the washings. Remove the cap, place the cup on its side in a 250-milliliter (ml) beaker, wash the material adhering to the underside of the cap into the beaker with a stream of water from a wash bottle, cover with a watch glass and cautiously add 100 ml of hot distilled water. When the material in the cup is dissolved remove the cup and rinse any remains of the fusion into the beaker. Make the solution neutral to litmus paper with hydrochloric acid allowing sufficient time between the additions of the acid for the completion of the reaction. Add 1 ml of acid in excess. Filter the solution and wash with hot water. Make up the filtrate to 200 ml with distilled water. Heat the solution to boiling and add slowly to the boiling liquid 10 ml of a 10 percent solution of barium chloride. Continue boiling for 15 minutes and allow to stand for 2 hours at a temperature just below the boiling point. Filter the precipitate through a tared filtering crucible and wash well with hot water. Dry the crucible and contents in an oven and ignite at a temperature of 850 ± 50°C. Cool in a desiccator and weigh. Calculate the increase in weight as percentage of sulfur in the sample on a moisture-free basis as follows:

$$\text{Percentage of sulfur} = \frac{13.74A}{W}$$

where: A = Weight of barium sulfate, gm

W = Weight of dry sample, gm

4.7.3.1 Alternate ignition procedure. Sulfur content may be determined alternatively by the use of a Parr type oxygen bomb utilizing oxygen from an external source as follows:

4.7.3.1.1 Apparatus.

Bomb - Parr instrument No. 1101 self-sealing oxygen combustion bomb model BB or approved equivalent with double valve.

Cup - Stainless steel, capacity 6.5 ml.

Firing wire - Iron-nickel chromium alloy wire 0.0063 inch nominal diameter

4.7.3.1.2 Ignition. The firing wire shall be clean and so arranged that it will touch the sample when the cup is placed in position in the bomb. Place 5 ml of distilled water in the bomb to saturate with water vapor the oxygen used for combustion. Transfer an accurately weighed portion of approximately 0.4 gm of the sample to the cup. The cup shall be placed in the bomb and the bomb closed. Screw on the cover of the bomb firmly to avoid leaks under pressure. Admit oxygen from the supply cylinder slowly to avoid blowing the sample from the cup. Cease the addition of oxygen after a minimum gas pressure of 40 atmospheres has been reached. Make the electrical connections, place the bomb in a bucket of cold water and ignite the sample. After 10 minutes remove the bomb from the water and release the pressure at a uniform rate such that the operation requires at least 1 minute. Open the bomb and examine for traces of unburned sample. If traces of unburned sample are found, the determination shall be discarded.

4.7.3.1.3 Solution, precipitation, and calculation. The solution and precipitation shall be carried out, and the calculation of percent sulfur shall be made as specified in paragraph 4.7.3.

4.7.3.2 Acceptance criteria. For the lot represented to pass the sulfur test, the value obtained for the percent sulfur shall not be more than the value specified in 3.4.1.

4.7.4 Acetone extract. Transfer an accurately weighed portion of approximately 2 gm of the sample to a 200 ml volumetric flask and add 100 ml of acetone. Heat to boiling, cool, fill to the mark with acetone and mix. Permit the residue to settle, draw off approximately one-half of the supernatant liquid and filter through a dry paper. Discard

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the first 10 ml of the filtrate and transfer 100 ml of the clear filtrate to a tared dish. Evaporate the acetone at a temperature not above 75°C and then heat the dish and contents for 15 minutes in an oven at 105 to 110°C. Cool in a desiccator and weigh. Calculate the weight of residue as percentage of acetone extract in the sample on a moisture-free basis.

4.7.4.1 Acceptance criteria. For the lot represented to pass the acetone test, the value obtained for percent acetone shall not be more than the value specified in 3.4.1

4.7.5 Organic dyes. Determine visually the presence or absence of organic dyes by examining the acetone and water extracts.

4.7.5.1 Acceptance criteria. For the lot represented to pass the organic dyes test, the value obtained for percent organic dyes shall not be more than the value specified in 3.4.1.

4.7.6 Ash content. Weigh accurately approximately a 1-gm sample into a previously ignited, tared 30 ml porcelain crucible and heat gently in a hood to volatilize all organic matter. Transfer the crucible to a muffle furnace at 900 ± 50°C until a constant weight is reached (4 to 5 hours). Cool in a desiccator until a constant weight is obtained and reweigh. Calculate the percent ash in the sample on a moisture free basis as follows:

$$\text{Percent ash} = \frac{B \times 100}{A}$$

where: A = Weight of sample, gm

B = Weight of ash, gm

4.7.6.1 Acceptance criteria. For the lot represented to pass the ash test, the value obtained for the percent ash shall not be more than the value specified in 3.4.1.

4.7.7 Acidity (hydrogen ion concentration, pH). Perform acidity test in accordance with the procedure given in Test Method D1512-60 "pH Value of Carbon Black" (Part 28 of ASTM, page 711).

4.7.7.1 Acceptance criteria. For the lot represented to pass the acidity test, the value obtained for percent pH shall be within the range specified in 3.4.1.

4.7.8 Water soluble material. Transfer an accurately weighed portion of approximately 5 gm of the sample to a beaker and add 150 ml of distilled water. Boil for 15 minutes, cool and transfer to a 200-ml volumetric flask. Dilute to the mark with distilled water and mix. Let settle, draw off approximately one-half of the supernatant liquid and filter through a dry paper. Discard the first 10 ml of the filtrate and transfer 100 ml of the clear filtrate to a tared dish. Evaporate to dryness, cool in a desiccator and weigh. Calculate the weight of residue as percentage of water soluble material in the sample on a moisture-free basis.

4.7.8.1 Acceptance criteria. For the lot represented to pass the water soluble test, the value obtained for percent water soluble material shall not be more than the value specified in 3.4.1.

4.7.9 Coarse particles. Prepare a stock solution of dispersing agent in water using as high a concentration of dispersing agent as possible without losing fluidity, and filter through coarse filter paper. (Trition 720, Darvan No. 1, Nacconol, or any other dispersing agent suitable for carbon black may be used.) Place a 10-gm portion of the sample in a 600-ml beaker. Add enough dispersing agent solution to form a heavy paste, and mix well to thoroughly incorporate the carbon black. Dilute the mixture with water to a volume of approximately 300 ml, and then pour through a U.S. Standard No. 325 sieve in accordance with RR-S-366. Wash the material on the sieve with a steady, gentle stream of water which first has been passed through a U.S. Standard No. 325 sieve. Using a small camel's-hair brush, brush the material through the sieve, running water through at the same time. Continue this operation until no more carbon black passes through the sieve. Once or twice during the screening, it may be helpful to remove the sieve from under the running water, add a drop or two of the stock dispersing agent solution,

thoroughly spread it over the sieve with the brush, and continue the washing and brushing. Dry the sieve in an oven at 100 to 105°C for 1 hour, cool and weigh the residue. Calculate the weight of residue as percentage of coarse particles in the sample on a moisture-free basis. Retain the residue for the determination of grit.

4.7.9.1 Acceptance criteria. For the lot represented to pass the coarse particles test, the value obtained for percent of coarse particles shall not be more than the value specified in 3.4.1.

4.7.10 Grit. Transfer the residue (if any) obtained as specified in 4.7.9 to a piece of smooth white paper. Gently rub the residue with the finger until the paper is no longer blackened by the remaining dispersible carbon black. Transfer the grit particles to a tared weighing dish; weigh and calculate the weight of the particles as percentage of grit in the sample on a moisture-free basis.

4.7.10.1 Acceptance criteria. For the lot represented to pass the grit test, the value obtained for percent grit shall not be more than the value specified in 3.4.1.

4.7.11 Volatile material. Perform volatile test in accordance with the procedure given in Test Method D1620-60 "Volatile Content of Carbon Black" (Part 28 of ASTM, page 767).

4.7.11.1 Acceptance criteria. For the lot represented to pass the volatile test, the value obtained for percent volatile material shall not be more than the value specified in 3.4.1.

4.7.12 Iodine value. Perform iodine test in accordance with the procedure given in Test Method D1510-65 "Iodine Absorption Number of Carbon Black" (Part 28 of ASTM, page 706).

4.7.12.1 Acceptance criteria. For the lot represented to pass the iodine test, the value obtained for percent iodine shall be within the range specified in 3.4.1.

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4.7.13 Benzene solubles. Transfer an accurately weighed sample of 12 gm to a double-thickness paper Soxhlet-extraction thimble, 94 by 33 millimeters (mm). Insert a plug of glass wool in the top of the thimble to prevent loss by splattering, of carbon black. Extract with 150 ml of reagent-grade benzene (C_6H_6) until no further discoloration appears in the benzene passing through the thimble. Transfer a portion of the benzene extract from the extraction vessel to an accurately weighed flat-bottom, glass evaporating dish, 90 by 50 mm. Evaporate to a small volume in a well ventilated hood. Transfer and evaporate successive portions of the benzene extract from the extraction flask until none remains; finally rinse the extraction flask with about 15 ml of fresh benzene. Add the rinsings to the evaporating dish. Evaporate the benzene completely. Dry the residue at 100-110°C to a constant weight.

$$\text{Percent benzene solubles} = \frac{100\ (A-B)}{W}$$

where: A = Weight of evaporating dish plus residue, gm.

B = Weight of evaporating dish empty, gm.

W = Weight of sample, gm.

WARNING

Benzene, a highly toxic chemical, is also flammable; its vapors form explosive mixtures with air.

4.7.13.1 Acceptance criteria. For the lot represented to pass the benzene solubles test, the value obtained for percent benzene solubles shall not be more than the value specified in 3.4.1.

4.7.14 Bulk density. Transfer an accurately weighed portion of approximately 10 gm of the sample to a 100 ml graduated glass stoppered precision grade cylinder. Drop the cylinder vertically from a height of 2-1/2 inches onto a solid surface until carbon black level no longer packs. Level the surface of the specimen in the cylinder by gently tapping and read the volume in millimeters. Calculate the bulk density as follows:

$$\text{Bulk density} = \text{gm per ml} = \frac{W}{V}$$

where: W = Weight of specimen, gm.

V = Volume occupied by the specimen, ml.

4.7.14.1 Acceptance criteria. For the lot represented to pass the bulk density test, the value obtained for the bulk density shall be within the range specified in 3.4.1.

4.7.15 Oil absorption. Perform oil absorption test in accordance with the procedure given in Test Method D1483-60 "Oil Absorption of Pigments by Gardner-Coleman Method" (Part 20 of ASTM, page 670).

4.7.15.1 Acceptance criteria. For the lot represented to pass the oil absorption test, the value obtained for the percent oil absorption shall be within the range specified in 3.4.1.

4.8 Packing and marking. Determine that all packing and marking conforms to section 5 of this document.

5. PREPARATION FOR DELIVERY.

5.1 Preservation and packaging. Not applicable (unless specified in the contract or purchase order).

5.2 Packing.

5.2.1 Level A. Not applicable.

5.2.2 Level B. Not applicable.

5.2.3 Level C. The material shall be packaged as directed in the contract to afford protection against damage during direct shipment from the supply source to the first receiving activity for immediate use. Containers shall comply with common carrier regulations applicable to the mode of transportation to be used. See 6.2.

5.3 Marking.

5.3.1 Special markings. Not applicable.

5.3.2 Normal markings. In addition to the markings required by contract or purchase order, unit packages and shipping containers shall be marked in accordance with the requirements of MIL-STD-129.

6. NOTES.

6.1 Intended use. Carbon black covered by this document is intended for use as an ingredient in ammonium-nitrate-based solid propellants.

6.2 Ordering data. Procurement documents should specify, but not be limited to, the following information:

(a) Title, number and date of this document.

(b) Applicability of the preproduction sample, (see 3.1).

(c) Invocation of MIL-Q-9858.

(d) Type and size of shipping container.

6.3 Definition. A batch is defined as that quantity of material which has been subjected to one or more chemical or physical processes (or combinations thereof) intended to produce a desired product having substantially uniform characteristics. The final step in the processing must have treated the entire contents of the batch at one time.

6.4 Safety and health warning. Handling of the chemicals specified herein shall be in accordance with suitable safety and health precautions.

6.5 Acceptable product. An acceptable product under this document is Channel Carbon Black, Texan E, manufactured by the Sid Richardson Carbon Company, Akron, Ohio.

Custodian:
NASC 52021E

Preparing Activity:
NWC/China Lake, California